



Harnessing surface wrinkling in film-substrate system by precisely controlling substrate modulus



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ABSTRACT

Wrinkled surfaces with controlled morphologies and sizes are quite useful for a wide range of applications including electronics, optics, bionics and metrology. Here we report on the controllable formation of surface wrinkling in metal films deposited on compliant substrates by precisely controlling substrate modulus. The wrinkling patterns are categorized as two classes of distinct features: G1 and G2 wrinkling. The G1 wrinkling emerges on very soft surfaces, originating from the surface instability of soft substrate during sputtering. The G2 wrinkling emerges on relatively stiff surfaces, originating from the thermal compression of film-substrate system after deposition. The size of G1 wrinkling decreases with increasing the substrate stiffness but is insensitive to the film thickness. The size of G2 wrinkling increases with increasing the film thickness or decreasing the substrate stiffness. The formation mechanisms and evolutionary behaviors of the wrinkling patterns have been analyzed based on the stress model. The hydrophobicity of the wrinkled surfaces is also presented in this work.

1. Introduction

Wrinkling is a very common phenomenon in film-substrate bilayer system when subjected to a compression. Many previous studies have shown that the wrinkled surfaces are quite useful for a wide range of applications including flexible electrodes [1], stretchable electronics [2], diffraction gratings [3], optical smart window [4], elastomeric microlens array [5, 6], deployable smart particles [7, 8], tissue engineering [9], surface hydrophobicity [10], adhesion [11], sensor [12], nanogenerator [13], etc. Many strategies have been developed to control the wrinkle morphology, orientation and size for practical applications. It is well known that the wrinkle morphology and orientation are strongly dependent on the stress field in the film. An isotropic stress field usually leads to randomly oriented labyrinth wrinkling [14, 15]. A unidirectional stress generates parallel stripe wrinkles perpendicular to the stress direction [1, 3, 4, 9, 16]. Sequentially releasing the biaxial stresses can result in highly ordered herringbone wrinkling [17, 18]. In addition, film impurity, edge effect, gradient material and restricted deformation would change the stress field temporally or spatially, leading to more complex wrinkling patterns [14, 19, 20].

On the other hand, the wrinkle size is strongly dependent on the structures and properties of film-substrate system. According to the continuum elasticity theory, the energy minimization of the system leads to a balanced wrinkle wavelength expressed as [14]

$$\lambda = 2\pi h \left[\frac{E_f(1 - \nu_s^2)}{3E_s(1 - \nu_f^2)} \right]^{1/3}, \quad (1)$$

where h is the film thickness, E_f and E_s are Young's moduli of film and substrate, ν_f and ν_s are Poisson's ratios of film and substrate, respectively. It is clear that the wavelength is directly proportional to the film thickness when the material parameters are constant, which has been confirmed by many experimental observations [3, 18–20]. The wrinkle size can be also tailored by controlling the modulus ratio of film to substrate. The previous studies showed that the substrate (e.g., Polydimethylsiloxane, PDMS) modulus can be conveniently tuned by changing the ratio of crosslinker to prepolymer [21, 22]. Our group successfully prepared a PDMS substrate with gradient modulus by tuning the temperature field during the curing process [23, 24]. However, precisely controlling the modulus of compliant substrate remains a challenge due to the complex relationship between the elastic modulus and the crosslinker content or curing temperature. In this work, we develop a facile technique to tune the substrate modulus by simple mixture of low stiffness gels into high stiffness elastomers. The wrinkling patterns of subsequently deposited metal films can be well harnessed by the substrate modulus and are good consistent with the theoretical predications. The present study can promote better understanding of the effect of substrate modulus on wrinkling patterns and develop a facile technique to fabricate various controllable wrinkled

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surfaces for practical applications.

2. Experimental details

The compliant substrates with different moduli were prepared by mixing low stiffness dielectric gels (Sylgard 527) into high stiffness elastomers (Sylgard 184 PDMS). Both products were commercially available organics, purchased from Dow Corning. The mixtures had stable mechanical, physical, chemical and surface properties. Firstly the prepolymers and curing linkers of PDMS and dielectric gel were mixed with 10:1 and 1:1 weight ratios, respectively. Then the liquid PDMS and liquid gel were mixed with various ratios according to volume, namely PDMS content w . After degassing in a vacuum chamber, the mixtures were spin-coated onto pre-cleaned $\sim 10 \times 10 \text{ mm}^2$ glass sheets with the rotation speed and rotation time of 5000 rpm (revolutions per minute) and 30 s, respectively. Subsequently, the samples were baked at 80°C for 3 h to crosslink the mixture completely. After that, metal films were deposited on the compliant substrates with different moduli simultaneously by direct current sputtering technique. It was found that various metals (including molybdenum, iron, nickel, etc.) lead to similar experimental results. The experimental data presented in this paper were obtained from molybdenum films. The molybdenum target was a piece of disk with 60 mm diameter and 3 mm thickness. The sputtering power was fixed at 50 W under the argon gas pressure of 0.5 Pa. The deposition rate was $\sim 15 \text{ nm/min}$ and the deposition time was controlled by a computer (in the range of 0.5–25 min). The surface morphologies were detected by an optical microscopy (Olympus BX41) and an atomic force microscopy (AFM, XE-100E, PSIA) operated in noncontact mode.

3. Results and discussion

Fig. 1 shows the morphological phase diagram with respect to the film thickness h (vertical coordinate) and the PDMS content w (horizontal coordinate) taken by the optical microscopy. We find that wrinkling patterns generally form on the sample surfaces except for the case of very small film thickness and very soft substrate, where

localized creasing can be seen clearly. The wrinkling patterns are categorized as two classes of distinct features: G1 and G2 wrinkling. The G1 wrinkling is favorable for lower PDMS content ($< 20\%$) whereas the G2 wrinkling is dominant for higher content ($\geq 20\%$). As the PDMS content increases, the sizes of both G1 and G2 wrinkling decrease obviously when the film thickness is given. If the PDMS content is fixed, the size of G2 wrinkling increases obviously with increasing the film thickness while that of G1 wrinkling is insensitive to the film thickness.

To detect more details of these two classes of wrinkling patterns, they were taken by the atomic force microscopy (AFM), as shown in Fig. 2. The comparison of sectional profiles for different PDMS contents is shown in Fig. 3(a). It is clear that the G2 wrinkling possesses a well-defined wavelength (or period), but the G1 wrinkling does not. In addition, localized creasing structure can be seen clearly among the G1 wrinkling (see the arrows in Fig. 2), demonstrating different formation mechanisms for G1 and G2 wrinkling. To further understand the evolution behavior of the wrinkling patterns, we measured the wrinkle wavelengths for different film thicknesses and different PDMS contents, as shown in Fig. 3(b). Fig. 3(c) shows the enlarged view of G1 wrinkling. We find that the wavelengths of both G1 and G2 wrinkling decrease steadily with increasing the PDMS content. The wavelength of G2 wrinkling is strongly dependent on the film thickness while that of G1 wrinkling is almost independent of film thickness. Fig. 3(d) shows the dependence of the wrinkle wavelength λ on the film thickness h for pure PDMS ($w = 100\%$) and pure dielectric gel ($w = 0\%$) substrates. It is clear that the wavelength on pure PDMS substrate increases linearly with the film thickness whereas that on pure gel substrate is almost unchanged with the film thickness.

According to the previous studies, the sputtering atoms with high kinetic energy can penetrate into the very soft substrate, leading to the surface instability and pattern formation during the sputtering process [24, 25]. At the very early stage of film deposition, the penetration effect (also induced compressive stress) is very high and thus networked creasing (not wrinkling) is observed. As the film thickness or substrate stiffness increases, the penetration-induced compressive stress decreases and thus homogenous G1 wrinkling forms. The G1 wrinkling is resulted from the surface instability of soft substrate owing to atom

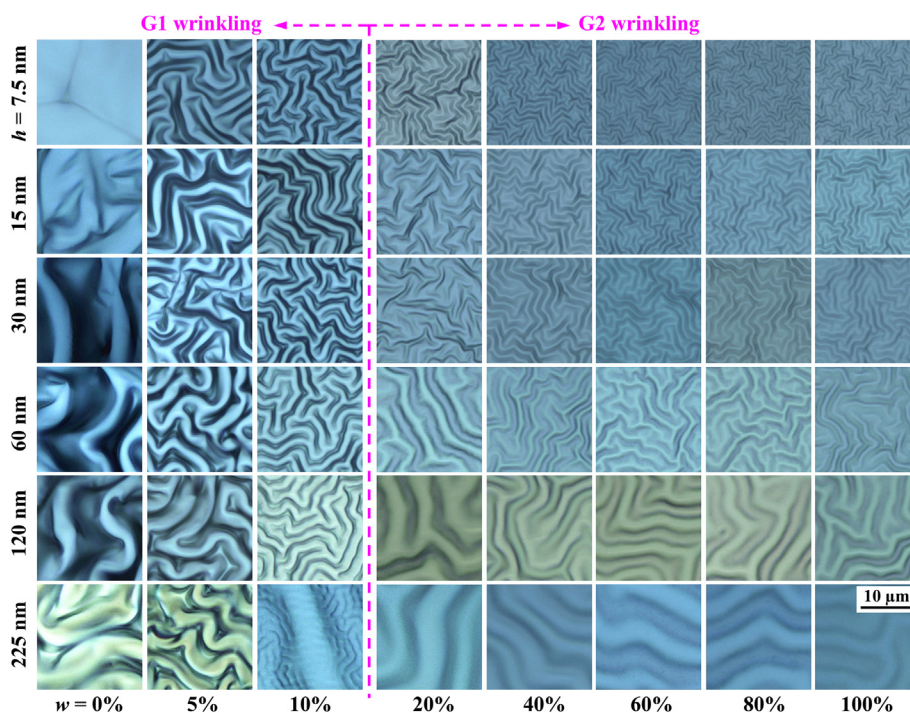


Fig. 1. Optical micrographs showing the morphological phase diagram with respect to the film thickness h and the PDMS content w . All images have the same size of $20 \times 20 \mu\text{m}^2$. Note that two classes of distinct wrinkling are observed in the samples, depending on the PDMS content.

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